National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 8k

Bessemer Steel (Simulated) 0.1 % Carbon

(In Cooperation with ASTM International)

This Standard Reference Material (SRM®) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 8k consists of a bottle containing approximately 150 g of fine millings sized between 0.50 mm (No. 35 sieve) and 1.18 mm (No. 16 sieve).

Certified values for four elements in SRM 8k are listed in Table 1. Reference values for six elements are listed in Table 2. For all elements, values are reported as mass fractions [1]. Test methods used to determine these elements are identified in Table 3.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. A certified value is the present best estimate of the "true" value based on the results of analyses performed at NIST and cooperating laboratories using the test methods listed in Table 3. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence interval [2] and is calculated according to the method in the ISO and NIST Guides [3].

Reference Values: Reference values are non-certified values that are the present best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all components of uncertainty. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence interval [2] and is calculated according to the method in the ISO and NIST Guides [3].

Expiration of Certification: The certification of this SRM is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). However, the certification will be nullified if the SRM is damaged or contaminated.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The overall coordination of technical measurements leading to certification was performed under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by J.H. Yen of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 20 April 2006

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

To relate analytical determinations to the values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The millings do not require preparation prior to weighing and dissolution. The material should be stored in its original container in a cool, dry location.

Table 1. Certified Values for SRM 8k Bessemer Steel (Simulated)

Constituent	(Mass Fraction) (%)	Uncertainty (Mass Fraction) (%)
Manganese	0.5046	0.0083
Copper	0.0200	0.0027
Chromium	0.0467	0.0027
Vanadium	0.0145	0.0011

Table 2. Reference Values for SRM 8k Bessemer Steel (Simulated)

Constituent	(Mass Fraction) (%)	Uncertainty (Mass Fraction) (%)
Carbon	0.0806	0.0014
Phosphorus	0.0956	0.0023
Sulfur	0.0775	0.0050
Silicon	0.0576	0.0083
Nickel	0.1174	0.0045
Molybdenum	0.0397	0.0036

PLANNING, PREPARATION, TESTING, ANALYSIS

For complete documentation, the details of planning, preparation, testing, and analysis for SRM 8j are included here.

SRM 8k was produced in cooperation with ASTM International Committee E01 Analytical Chemistry of Metals, Ores and Related Materials. The material for the preparation of this SRM was prepared by the Carpenter Technology Corporation (Reading, PA). This particular batch of steel was held in reserve after the certification of SRM 8j.

Certification of SRM 8k is based on a careful comparison of the reserve material to SRM 8j using a measurement procedure based on high-performance inductively coupled plasma optical emission spectrometry (ICP–OES) and the Bonferroni method of simultaneous statistical inference [4]. Except for Ni, all constituents were shown to be of the same composition in both materials. Due to a statistically significant difference between materials, Ni was determined using the Inductively Coupled Plasma Optical Emission Spectrometry measurements with SRM 8j as the calibrant.

The technical and support aspects involved in the original preparation and analysis of Standard Reference Material 8j were coordinated through the National Bureau of Standards (NBS) Office of Standard Reference Materials by R.E. Michaelis.

Homogeneity testing was performed at NBS by optical emission spectrometric analysis – J.A. Norris; C/S analysis – B.I. Diamondstone; and selected chemical analyses – R.K. Bell (Assistant Research Associate, ASTM/NBS Research Associate Program).

Cooperative analyses for certification were performed in the following laboratories:

National Bureau of Standards, Institute for Materials Research, Analytical Chemistry Division; S.A. Wicks, R.K. Bell, and E.R. Deardorff.
Inland Steel Company (East Chicago, IN) R.W. Bley.
Bethlehem Steel Corporation (Sparrows Point, MD) R.H. Rouse.
U.S. Army Materials and Mechanics Research Center (Watertown, MA) F.P. Valente.

Table 3. Analytical Methods Used to Determine the Element

Element	Methods
Carbon	Combustion – Infrared
Manganese	Persulfate Arsenite; $NaAsO_2$ Potentiometric Titration; KIO_4 Photometric Method
Phosphorus	Molybdate-Hydrazine Sulfate Photometric Method; Alkali-Molybdate; $Mg_2P_2O_7$ Gravimetric Method
Sulfur	Combustion – KIO_3 Titration
Silicon	Perchloric Acid Double Dehydration; Silicomolybdate Photometric Method
Copper	Diethyldithiocarbamate Photometric Method; Neocuproine Photometric Method
Nickel	Dimethylglyoxime Gravimetric Method; Dimethylglyoxime Spectrophotometric Method
Chromium	$NaHCO_3$ Hydrolysis – Peroxydisulfate Oxidation – Potentiometric Titration with $Fe(NH_4)_2(SO_4)_2$; Diphenylcarbazide Photometric Method; Atomic Absorption Spectrophotometry; Peroxydisulfate Oxidation – $Fe(NH_4)_2(SO_4)_2$ Reduction – $KMnO_4$ Titration; Peroxydisulfate Oxidation Amperometric Titration with $Fe(NH_4)_2(SO_4)_2$
Vanadium	Atomic Absorption Spectrophotometry; $NaHCO_3$ Hydrolysis – HNO_3 Oxidation – Potentiometric Titration with $Fe(NH_4)_2(SO_4)_2$; $Fe(NH_4)_2(SO_4)_2$ Reduction – $KMnO_4$ Titration; $KMnO_4$ – KNO_2 Urea – $Fe(NH_4)_2(SO_4)_2$
Molybdenum	Thiocyanate-$SnCl_2$ Spectrophotometric Method

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).
[2] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).
[3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.
[4] Miller R.; *Simultaneous Statistical Inference*; Springer-Verlag, New York (1981).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.